OIL EMULSION SEPARATION USING FORWARD OSMOSIS AND ITS PERFORMANCE EVALUATION

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OIL EMULSION SEPARATION USING FORWARD OSMOSIS AND ITS PERFORMANCE EVALUATION

by

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Melvin Raj

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LIST OF SYMBOLS

	Symbol	Unit
A	Membrane area	m^2
A	Pure water permeability coefficient	$L/m^2.hr.bar$
B	NaCl permeability coefficient	$L/m^2.hr$
c_f	Concentration of oil in feed solution	ppm
c_p	Concentration of oil in draw solution	ppm
D	Dye concentration at time, t	m^2/hr
J_w	Flux	L/m2.hr
R	Rejection rate	-
S	Structural parameter	mm
t	Time taken for permeation	hr
V	Volume of permeate	L
$\pi_{D,b}$	Osmotic pressure of bulk draw solution	bar

LIST OF ABBREVIATIONS

CP	Concentration polarization
DS	Draw solution
ECP	External concentration polarization
FO	Forward osmosis
FS	Feed solution
ICP	Internal concentration polarization
MF	Microfiltration
NF	Nanofiltration
PRO	Pressure retarded osmosis
ppm	Parts per million
RO	Reverse osmosis
SEM	Scanning electron microscopy
TFC	Thin film composite
UF	Ultrafiltration
UV	Ultraviolet

PEMISAHAN EMULSI MINYAK MENGGUNAKAN MEMBRAN OSMOSIS HADAPAN DAN PENILAIAN PRESTASINYA

ABSTRAK

Dalam kajian ini, pemisahan air daripada air yang dihasilkan sintetik dan emulsi minyak dalam air melalui osmosis ke hadapan telah dijalankan. Membran yang digunakan dalam kajian ini adalah membran komposit filem tipis (TFC) dengan lapisan poliamida aktif. Kesan kepekatan minyak makanan yang berbeza (50ppm, 100pp, dan 500ppm) pada prestasi membran dipelajari. Telah didapati bahawa fluks air menurun apabila kepekatan minyak suapan meningkat kerana pembentukan lapisan kek pada permukaan membran. Fluks permeat emulsi minyak dalam air adalah lebih tinggi daripada air yang dihasilkan sintetik kerana kehadiran garam laut dalam air yang dihasilkan sintetik yang memberikan tekanan osmotik yang lebih tinggi. Fenomena pencemaran membran diperhatikan dan dianalisis dengan menggunakan mikroskop elektron scanning (SEM). Membran TFC yang digunakan dalam kajian ini memberikan kadar penolakan minyak yang berkesan sebanyak 99.2% dan kadar pencemaran yang minima. Nilai fluks yang diperoleh untuk air yang dihasilkan sintetik dan emulsi minyak dalam air adalah 0.001295 $m^3/m^2.h$ and 0.004078 $m^3/m^2.h$.

OIL EMULSION SEPARATION USING FORWARD OSMOSIS MEMBRANE AND ITS PERFORMANCE EVALUATION

ABSTRACT

In this study, the separation of water from synthetic produced water and oil-in-water emulsion via forward osmosis was carried out. The membrane used in this study was thin film composite (TFC) membrane with an active polyamide layer. The effect of different feed oil concentrations (50ppm, 100pp, and 500ppm) on the membrane performance was studied. It was found that the water permeate flux decreased when the feed oil concentration was increased due to the formation of cake layer on the membrane surface. The permeate flux of oil-in-water emulsion is higher than that of synthetic produced water due to the presence of sea salt in the synthetic produced water that gives a higher osmotic pressure. The fouling phenomena of the membrane is observed and analysed using scanning electron microscope (SEM). The TFC membranes used in this study gave an effective oil rejection rate of 99.2% with minimal fouling tendency. The flux values obtained for synthetic produced water and oil-in-water emulsion were 0.001295 $m^3/m^2.h$ and 0.004078 $m^3/m^2.h$ respectively.

CHAPTER ONE

INTRODUCTION

1.1 Research Background

In recent years, the exponential growth of the world population along with other factors like rapid industrialization and pollution has resulted in shortage of fresh and clean water supply (Akther, et al., 2015; Zhao, et al., 2016). Water supply is largely required not only for human consumption but also for agricultural needs and product development in many industries (Duong & Chung, 2014). Therefore, the need for water recycling and reusing has tremendously increased. Subsequently, a lot of effort has been put on researches to develop new technologies that can efficiently extract fresh water from seawater or wastewater sources (Zhao, et al., 2016).

Oily wastewater is one of the most commonly generated waste product in various industries such as the petrochemical, textile, leather and food industries, to name a few (Zhang, et al., 2017). In general, oily wastewater can exist in free, dispersed or emulsified form, depending on their size. Free oil is oil with droplet sizes greater than 150µm while dispersed oil has a size ranging from 20-150µm. Any droplet smaller than 20µm is known as emulsified oil (Duong & Chung, 2014; Han, et al., 2015; Cheryan, 1998). With industries booming worldwide, the discharge of oily wastewater has also increased, thus endangering the environment and causing major water and soil pollution (Ge, et al., 2017; Han, et al., 2015; Xu, et al., 2017). Hence, more emphasis has been placed on the treatment of oily wastewater.

The largest contributor to oily wastewater is the oil and gas industry (Susan, et al., 2017). The wastewater that is produced in this industry is known as produced water. Produced water contains various organic and inorganic substances such as oil components and dissolved minerals. It is estimated that about 250 million barrels of produced water is produced globally on a daily basis (Ahmadun, et al., 2009). This large amount of produced water, if discharged, will pollute the underground water source and soil and cause adverse environmental impacts. In order to combat this issue, environmental regulations have been implemented that allows for the oil and grease concentration in produced water to only be within 10-15 mg/L (Zhang, et al., 2014). Therefore, to comply with this regulation as well as reuse and recycling of produced water, industries have put more focus and importance on treating produced water.

Over the years, produced water has been treated via conventional methods such as gravity separation, coagulation, flocculation and air flotation. However, these techniques are only effective as far as free and dispersed oil particles are concerned. They are inefficient when it comes to removing stable emulsified oil particles. Due to this limitation, more advanced techniques were developed to improve the treatment process (Duong & Chung, 2014; Han, et al., 2015; Cheryan, 1998; Zhang, et al., 2017; Ge, et al., 2017; Zhang, et al., 2014).

Membrane based separation has been one of the most commonly used new technique as they are very much capable of removing the stable oil particles that goes unaffected by the traditional methods. Processes like microfiltration (MF), ultrafiltration (UF), nanofiltration (NF) and reverse osmosis (RO) have been commercially used in produced water treatment. They provide higher efficiency than conventional methods and also comes at a lower cost. Unlike conventional methods, these membrane processes do not require any chemical additives as well (Duong & Chung, 2014; Han, et al., 2015; Cheryan, 1998; Zhang, et al., 2017; Ge, et al., 2017; Zhang, et al., 2014).

In recent times, forward osmosis (FO) membrane technology is more preferably used than the other membrane processes. This is because unlike the other membrane processes that require an external hydraulic pressure, the FO process uses osmotic pressure as its driving force. As a result, FO runs with very little to no operational pressure, thus reducing the fouling tendency and increasing the life span of the membrane which in turns reduces the operational cost involved in replacing the membranes. FO also has a higher water recovery rate and higher salt and contaminant rejection rate than the other membrane processes (Duong & Chung, 2014; Han, et al., 2015; Cheryan, 1998; Zhang, et al., 2017; Ge, et al., 2017; Zhang, et al., 2014).

Thus, in this study, FO process was used in the recovery of water from the synthetic produced water. The performance of the FO process as well as the fouling behaviour of the membrane was studied at different oil concentrations.

1.2 Problem Statement

In treating produced water, it has been found that conventional methods like gravity separation, air flotation and coagulation were unable to remove the smaller and more stabilized oil molecules. Thus, attention has been shifted towards membrane-based separation. Membrane separation, however, comes with its own problems like fouling tendency. For this study, forward osmosis process has been chosen to recover water from the produced water. Compared to other membrane processes, forward osmosis do not require any external pressure to run, thus reducing the fouling issue. However, unstable oil emulsion will tend to foul the surface under high salinity conditions as well as poor hydrodynamic condition.

Factors like oil concentrations, draw solution concentrations and cross flow velocity also contributes to the overall efficiency of the separation process. Oil concentration level especially plays a big role in separation efficiency as the oil molecules present are usually the main cause of membrane fouling. In this experiment, feed solution with varying oil concentration has been used. The membrane performance and the efficiency of the forward osmosis process under these oil concentrations has been investigated.

1.3 Research Objectives

The objectives associated with this study are as follows:

1. To study the efficiency of forward osmosis membrane in separating water from oil-in-water emulsion and synthetic produced water
2. To study the effect of different feed concentrations on the separating efficiency of forward osmosis
3. To evaluate the fouling tendency of oil emulsion on the membrane surface

1.4 Scope of Study

In this experiment, self-synthesised thin film composite (TFC) membrane is used for forward osmosis separation. Three different concentrations (50ppm, 100ppm, 500ppm) of oil-in-water emulsion without sea salt and three different concentrations (50ppm, 100ppm, 500ppm) of synthetic produced water with 3.5wt% are used as feed solutions. The membrane performance in terms of flux and rejection will be compared for all three conditions.

1.5 Thesis Organization

This work is organized into five distinct chapters. The first chapter covers the research background, the problem statement and objectives of this study followed by the scope of this research. Chapter two comprises of a literature review on the research topic. The literature review contains further information and theoretical explanations regarding this project. The purpose of this study, as well as the challenges faced, is also more boldly stated in the literature review. Chapter three focuses on the materials and methods used in running this project. A list of the materials used, along with their function, is included. Furthermore, the experimental procedures involved are also described more thoroughly. Chapter four moves forward to the results and discussion area. The obtained raw data from the experiment are analysed and calculations are made to obtain required results. The discussions are mainly about these obtained results and their validity. Chapter five consists of the conclusion inferred from this experiment and appropriate recommendations that can improve future works. Finally, a list of references used to support the facts in this work is included after the completion of chapter five.

CHAPTER 2

LITERATURE REVIEW

2.1 Oily Wastewater

Oily wastewater is basically wastewater that contains a mixture of any kind of oil. Oil can exist in three different forms; free oil with droplet sizes greater than 150µm, dispersed oil with sizes ranging from 20-150µm and emulsified oil with sizes lesser than 20µm (Duong & Chung, 2014; Han, et al., 2015; Cheryan, 1998). Oily wastewater is largely produced from oil and gas drilling processes and the discharge amount continues to rise with the bloom in the oil and gas industry. The direct discharge of oily wastewater will result in water and soil pollution as well as endangering the health of living organisms (Han, et al., 2015). Therefore, the need for a proper and effective treatment technique has heightened.

2.2 Produced Water

Produced water is the largest waste stream released from the oil and gas industries with a global estimation of 3:1 volume-to-product ratio. In the year 2012, the United States alone generated 21.2 billion barrels of produced water (Dickhout, et al., 2017). Produced water contains a mixture of organic and inorganic compounds and their compositions can differ depending on the type of sources. In general, the major components of produced water includes dissolved and dispersed oil compounds, production chemical compounds, dissolved gases and dissolved minerals (Ahmadun, et al., 2009; Susan, et al., 2017). Produced water is known to be harmful for underground water sources and soil when discharged (Susan, et al., 2017). In some cases, it is known to change the physical and chemical properties of the soil itself (Padaki, et al., 2015). Therefore, proper treatment is required before it can be discharged.

2.3 Conventional Methods and its Flaws

Produced water is an oil-in-water emulsion, stabilized by surfactants. Due to this reason, conventional separation techniques that are normally used like gravity and skimming, flocculation, dissolved air flotation and coagulation will not be suitable in treating produced water. This is because these techniques are only efficient as far as free-floating oil and dispersed oil are concerned as they have bigger droplet sizes. Oil emulsions that are stabilized with surfactants have droplet sizes smaller than 20µm, thus requiring a longer residence time to rise to the top before gravity separation can be achieved. Furthermore, conventional methods only allow for 50-60% water recovery, which is not very commendable. Taking not of these shortcomings, advanced separation technologies, especially membrane separation, have been explored to maximise water reuse and recycling (Han, et al., 2015; Zhang, et al., 2014).

2.4 Membrane Technology for Treatment of Produced Water

Membrane separation technology has experienced a rapid growth in recent years and provides a promising solution in treating produced water due to its suitable pore sizes and its ability to remove emulsified oil particles without the aid of any de-emulsification process (Susan, et al., 2017). When compared to the conventional methods, membrane processes provide a higher efficiency in water recovery and also cost lesser. They do not require the addition of chemical additives as well (Ge, et al., 2017; Xu, et al., 2017). Some of the commonly applied membrane separation processes include microfiltration, ultrafiltration, nanofiltration, reverse osmosis.

One of the major disadvantage of all four of these membrane processes is that they require an external hydraulic pressure to run. Thus, these processes have a high operational pressure, making them more prone to membrane fouling. Membrane fouling not only greatly reduces the lifetime of the membrane but also causes an increase in the maintenance cost as the membranes will have to be replaced more frequently (Duong & Chung, 2014; Han, et al., 2015; Zhang, et al., 2014; Zhang, et al., 2017).

2.5 Forward Osmosis

Processes like MF, UF, NF and RO are superior to the conventional methods. However, it is undeniable that they still have certain flaws that have to be acknowledged. The shortcomings of these processes have therefore called for a more efficient and economical membrane process, paving the way for forward osmosis (FO). FO is also under the same membrane-based separation category as the other processes mentioned above. However, its different working principle has given it a considerable advantage. Unlike the other membrane processes that require an external hydraulic pressure, the FO process uses osmotic pressure difference across the membrane as its driving force in separating the water from contaminants. This means that a typical FO process operates at very little or no operational pressure at all, thus largely minimizing the fouling factor of membrane and promoting a longer membrane life. FO also has a higher water recovery and contaminant rejection rate compared to the other membrane processes, making it the more favourable option for water separation (Duong & Chung, 2014, Han, et al., 2015, Zhang, et al., 2014, Zhang, et al., 2017, Xu, et al., 2017).

2.6 Working Principle of Forward Osmosis

FO process consists of movement of water involving two solutions; feed solution (FS) and draw solution (DS) that are separated by a semi-permeable membrane as shown in Figure 2.1. FS is the solution with the lower osmotic pressure (higher water chemical potential) while DS is the solution with the higher osmotic pressure (lower water chemical potential). The driving force in this process comes from the osmotic pressure difference between these two solutions. Water is permeated from the FS to the DS, thus concentrating the FS and diluting the DS. The semi-permeable membrane is vital as it functions in allowing water molecules to pass through but rejects salt movement, thus making the separation possible. The permeated water is then subsequently recovered from the diluted DS, thus regenerating the DS so that it can be reused (Khorshidi, et al., 2016, Yang, et al., 2016, Cai & Hu, 2016, Linares, et al., 2014).

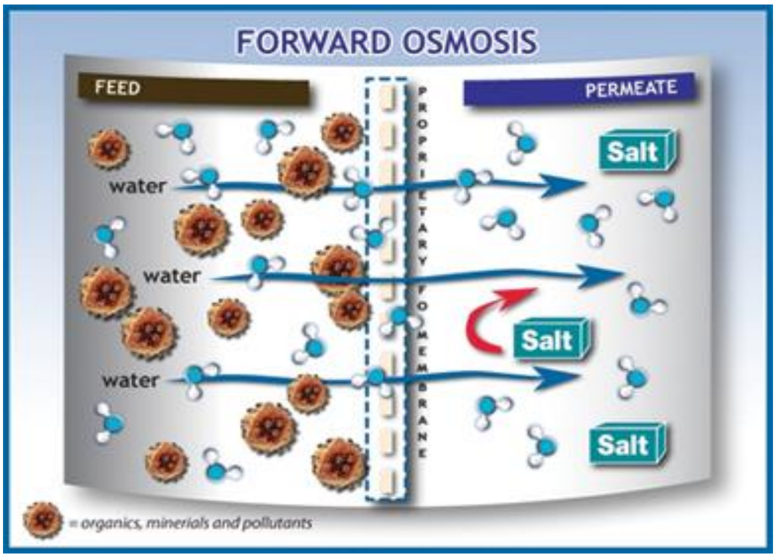

Figure 2.1: Diagram of forward osmosis process

Choosing a proper draw solution (DS) is important in order to ensure the FO process runs efficiently. Using a DS that is not suitable will result in complications such as low water flux and high reverse salt flux. The ideal DS for a FO process must satisfy certain criteria. The DS must be capable of generating a high osmotic pressure to provide a sufficiently strong driving force for the water molecules to be extracted from the feed solution. DS must also be chemically inert to the FO membrane so that it doesn’t tamper with the performance of the membrane. Another thing to take note is that DS should be easily separable from water so that the DS can be regenerated and pure water can be produced (Qasim, et al., 2017).

2.7 Forward Osmosis Application in Produced Water Treatment

Forward osmosis (FO) process has been largely applied in many researches on produced water treatment. In a study conducted by P.Li and S.S.Lim, they used thin film composite hollow fiber membranes in their FO process. The experiment was carried out with three different oil concentrations; 500ppm, 1500ppm and 3000ppm. Based on the results, it was clear that the FO process was applicable for all three concentrations. With a 1M NaCl as the draw solution, the obtained water flux for oil concentrations of 500ppm, 1500ppm and 3000ppm were 16.6 LMH, 14.9 LMH and 14.2 LMH respectively. It can be seen that with more oil particles present, the water flux decreases, indicating a possible fouling issue. The molarity of the draw solution was also varied and it was found that a higher salt concentration in the draw solution leads to a higher flux thanks to a higher osmotic driving force. The average oil rejection obtained in this FO process was up to 99.96%, indicating a very efficient process (Li, et al., 2014).

2.8 Challenges Faced by Forward Osmosis

It can be strongly said that FO is the most favourable membrane separation process compared to the other processes. However, no process is a 100% ideal. Despite being the best option, FO still comes with a few flaws that can affect the membrane performance and subsequently cause a drop in permeate flux.

2.8.1 Concentration Polarization

The main factor that dampens the membrane performance in FO is the occurrence of concentration polarization. Concentration polarization (CP) happens when there is a concentration difference at a membrane/solution interface due to the selective transfer of some species through the membrane. During FO process, some solutes tend to move towards the membrane. When these solutes get rejected and are unable to pass through the membrane, they tend to accumulate at the membrane surface, forming additional layers and obstacles for permeate water to pass through. This accumulation also causes the concentration of the solutes to be higher at the membrane surface than at the bulk of solution, thus forming the concentration difference that leads to CP. Two types of concentration polarization occurs in a typical FO process; external concentration polarization (ECP) and internal concentration polarization (ICP). ECP occurs on the surface of the active layer while ICP occurs inside the porous support layer (Akther, et al., 2015; Choi, et al., 2018).

ECP in itself can be further categorized into two types; concentrative ECP and dilutive ECP. When the solutes from the feed solution travels and accumulates at the surface of the active site, concentrative ECP is said to occur. Meanwhile, dilutive ECP occurs when the draw solution in contact with the permeate side of the membrane gets diluted by the incoming permeate water (Akther, et al., 2015). When these both processes occur, the effective osmotic pressure decreases, causing a reduction in the driving force and subsequently, a lower permeate flux. It is possible however, to diminish the effect of ECP in FO. This can be done by increasing the turbulence and shear rate of flow across the membrane (Choi, et al., 2018).

In FO process, the effect of ECP is considerably trivial when compared to ICP. Decrease in permeate flux in FO process is always mainly caused by ICP. Early studies show that ICP is capable of decreasing flux rate by over 80%. In FO mode, where the feed solution faces the active layer of membrane, dilutive ICP occurs. Dilutive ICP refers to when concentration of draw solution at the porous support layers are diluted by the transport of permeate water from the membrane. Concentrative ICP meanwhile occurs when operating in pressure retarded osmosis (PRO) mode, where the active layer faces the draw solution. Unlike ECP, an increase in turbulence or shear stress will not be able to overcome the effect of ICP due to the stagnant environment of the porous support layer. Therefore, ICP has become the main limiting factor in reduction of permeate flux (Akther, et al., 2015). Figure 2.2 shows the occurrence of ICP and ECP in forward osmosis.

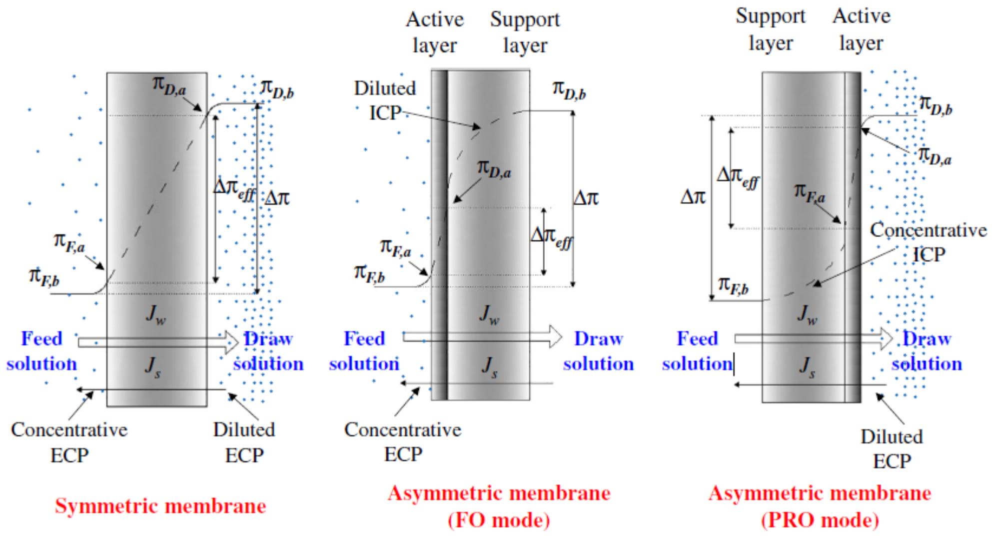

Figure 2.2: Diagram of internal concentration polarization (ICP) and external concentration polarization (ECP) *(Xu, et al., 2017)*

2.8.2 Reverse Solute Flux

Reverse solute flux is another occurrence that has crippled the performance of membrane in FO process. Reverse solute flux (RSF) is defined as the cross-membrane diffusion of solutes from draw solution to the feed solution. This leads to salinity build up in the feed solution and a reduction in effective osmotic driving force. The movement of solutes will also increase the fouling tendency of the membrane (Zou & He, 2017).

2.8.3 Membrane fouling

Membrane fouling is another phenomenon that affects all membrane processes. Membrane fouling occurs when solutes or particles accumulate at the membrane, as shown in Figure 2.3 below. This will not only reduce the water flux but also shorten the lifetime of the membrane. The operational cost will also be greater as the membrane will have to be cleaned more frequently or replaced with a new membrane altogether. In FO, the fouling is generally less notable compared to other membrane processes as they operate by osmotic driving force and not pressure-driven. Fouling in FO is also reversible and is capable of being cleaned by osmotic backwashing without the need of any specific chemical reagents (Akther, et al., 2015).

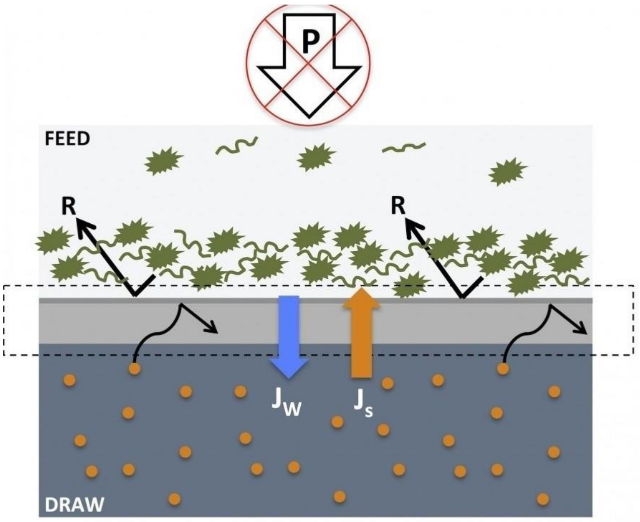

Figure 2.3: Diagram of membrane fouling in forward osmosis

CHAPTER 3

MATERIALS AND METHODS

3.1 Chemicals And Materials

Table 3.1 shows the list of chemical materials used in this study and their respective purposes.

Table 3.1: Table of chemicals and materials used in this work

MATERIALS	**PURPOSE**
Sodium chloride, NaCl **Supplier : Merck Millipore**	To prepare salt solution used as draw solution
Sea salt **Supplier : Sigma Aldrich**	To prepare the sea salt solution that is used as the feed solution
Hexadecane **Supplier : Sigma Aldrich**	Synthetic oil added to salt solution to form a synthetic produced water solution
Triton-X **Supplier : Sigma Aldrich**	A surfactant used to stabilize oil or hexadecane in the solution
Deionized water	- To prepare draw and feed solutions - To flush the forward osmosis unit - As part of pre-treatment of membrane
Ethanol, C_2H_5OH **Supplier : Fisher Scientific Sdn. Bhd.**	Membrane is immersed in 30vol% ethanol solution for 24 hours before its usage

3.2 Apparatus And Instruments

Table 3.2 shows the list of apparatus and their use in this study.

Table 3.2: Table of apparatus and instruments

APPARATUS	USE
Forward osmosis unit **Model : Cole-Parmer Instrument Company**	To run the forward osmosis process
Conductivity meter **Model : OHAUS Starter 300C**	To measure the conductivity of draw solution
Volumetric flask	To store the feed and draw solutions
Beakers	Used to mix and prepare solutions
Blender	To thoroughly mix the sea salt solution with surfactant and hexadecane
Spatula	To transfer salt crystals from their container into beakers
Sonicator **Model : Fisherbrand**	To sonicate oil emulsion and synthetic produced water
UV – visible spectrophotometer **Model : Agilent Technologies**	To measure the absorbance of samples
Universal oven **Model : Memmert Universal Oven**	To dry wet apparatus after use

3.3 Experimental Flow Chart

Figure 3.1 shows the basic overall experimental flow of this study.

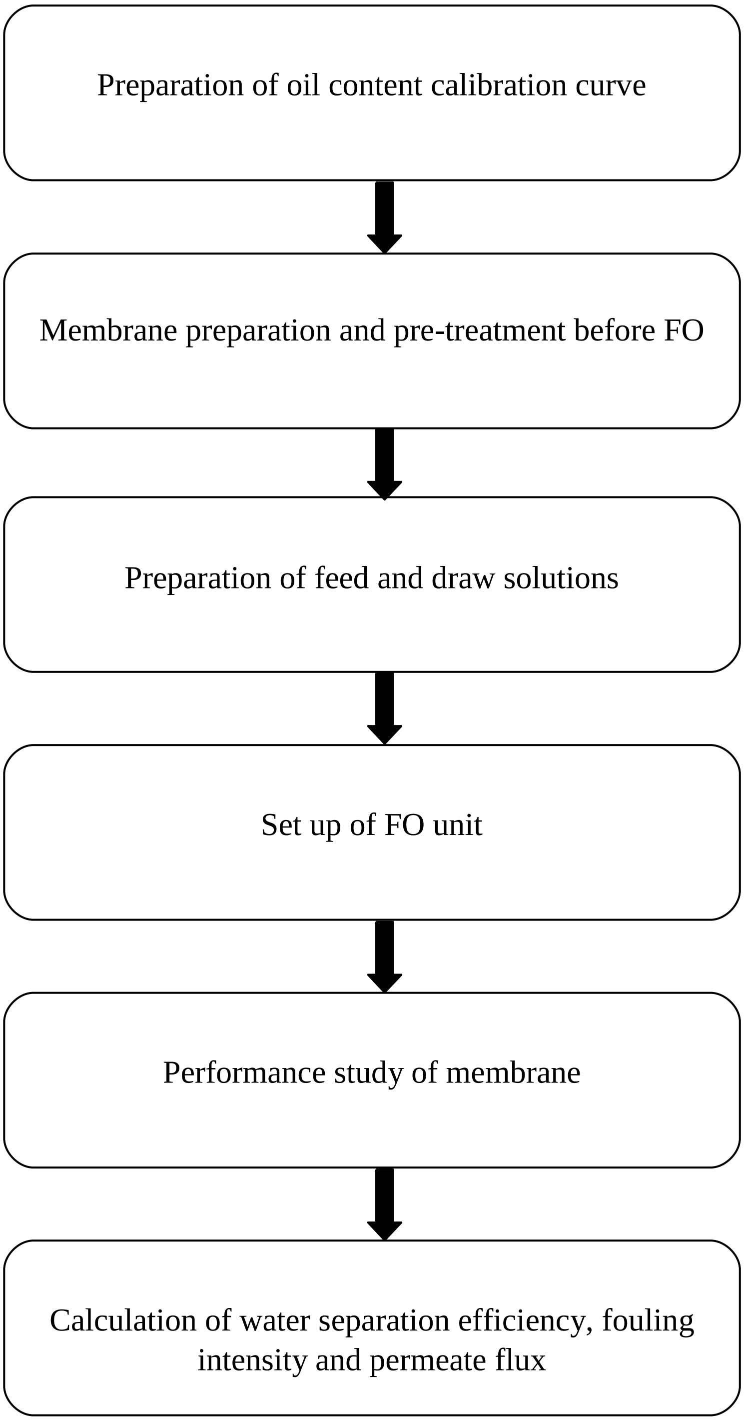

Figure 3.1: Overall experimental flow chart

The initial step of the experiment is the preparation of oil content calibration curve which will be used to find the oil concentration in draw solution after forward osmosis. This is followed by the preparation and pre-treatment of membrane. Then, the feed and draw solutions were prepared according to the required concentrations. Next, the FO unit is set up accordingly and the experiment is carried out. Finally, the membrane performance was analysed and important parameters such as the flux and rejection rate were calculated.

3.4 Calibration Curve

Calibration curve was constructed to determine the oil concentration in the sample. Different samples of draw solution with oil concentrations ranging from 0ppm – 300ppm were prepared. The absorptivity of each of the sample was then determined using the UV-Visible spectrophotometer at a wavelength of 200nm. Subsequently, a graph of absorbance versus concentration was plotted, which will be used later to determine the oil concentration in the final draw solutions.

3.5 Membrane Preparation and Pre-treatment

The thin film composite (TFC) used is a self-synthesised membrane. It consists of an active polyamide layer and a polysulfone support layer with a thickness of 150µm. Before the forward osmosis process was started, the prepared TFC membrane was pre-treated to enhance its efficiency so that a higher flux can be achieved during FO process. The first stage of pre-treatment was carried out by immersing the membrane in deionized water for 10 minutes. After this, the membrane was cut to the shape of the mould that is required to fit in the FO unit. An inaccurate membrane shape or size will result in the failure of the FO process. Once the required shape is achieved, the membrane is then immersed in a 30vol% ethanol solution for one day. This step is important because ethanol functions as a wetting agent in the membrane. After one day, the membrane was transferred to deionized water for another 10 minutes to remove the ethanol out, thus keeping the membrane consistently wet. After this step, the membrane is immediately placed in the FO unit and the process can be started.

3.6 Preparation of Feed and Draw Solutions

In this experiment, feed solutions with different oil concentration were prepared while the draw solution was kept constant at all times. The oil-in-water emulsion was prepared by adding the appropriate volume of hexadecane and surfactant Triton-X to a 1L solution of deionized water according to the concentration required. The solution was blended for 2 minutes and then sonicated for 5 minutes immediately after the oil and surfactant were added to ensure a well-mixed and stable solution.

In the case of produced water, feed solutions were prepared by mixing pure water, sea salt solution and oil emulsion. Sea salt solution was prepared by dissolving 32g of sea salt crystals in deionized water. Deionized water is added until the solution reaches a volume of 1L, with a molarity of 1M. In the preparation of synthetic produced water, a 1L sea salt solution was first prepared. The appropriate volume of hexadecane and surfactant Triton-X was then added to this sea salt solution according to the concentration required. Just like the preparation of oil-in-water emulsion, this solution was then immediately blended for 2 minutes and sonicated for 5 minutes to form a stable and well mixed solution.

For both oil-in-water emulsion and produced water, three different concentrations of oil was tested; 50ppm, 100ppm and 500ppm. Table 3.3 shows the volume of hexadecane and surfactant Triton-X that was added when preparing the different feed solutions with different concentrations.

Table 3.3: Volume of hexadecane and surfactant used for each feed oil concentration

Concentration (ppm)	**Volume of hexadecane (μL)**	**Volume of surfactant (μL)**
50	64.69	15
100	129.37	30
500	646.9	150

The draw solution used was the same for all the testing, which is a 1M NaCl solution with a volume of 1L. It was prepared by dissolving 58.44g of sodium chloride salt crystals in deionized water. Deionized water was then added to make up 1L solution with molarity of 1M.

3.7 Setup of Forward Osmosis Unit

In this experiment, a laboratory scale cross flow FO unit was used, as shown in Figure 3.2. Both the draw solution and the feed solution flows counter-currently at each side of the membrane. The membrane sheet was fixed in a membrane chamber within the membrane module, which was connected horizontally throughout the experiment. All the membrane sheets used have the same area of 42cm^2. The active layer was always set to face the side of the feed solution. Peristaltic pumps were used to pump both the feed and draw solutions towards the membrane module.

The FO unit also has two flow meters and pressure gauges to measure the flow rate and pressure of solutions entering and leaving the membrane module respectively. A portable conductivity meter was also placed in the draw solution to measure the change in conductivity of the solution over time. The conductivity reading was taken at every interval of 5 minutes. The draw solution was placed on an electronic balance to measure its change in mass, which will indicate the amount of permeate separated by membrane activity. The electronic balance was connected to a data acquisition system that displays the mass change on the computer. The interval for each recorded mass reading was set at 5 seconds. Each FO run duration was set at 1.5 hours. After each run was complete, the FO unit was flushed with deionized water for a duration of 15 minutes to ensure that the membrane will be in optimum condition for the next run.

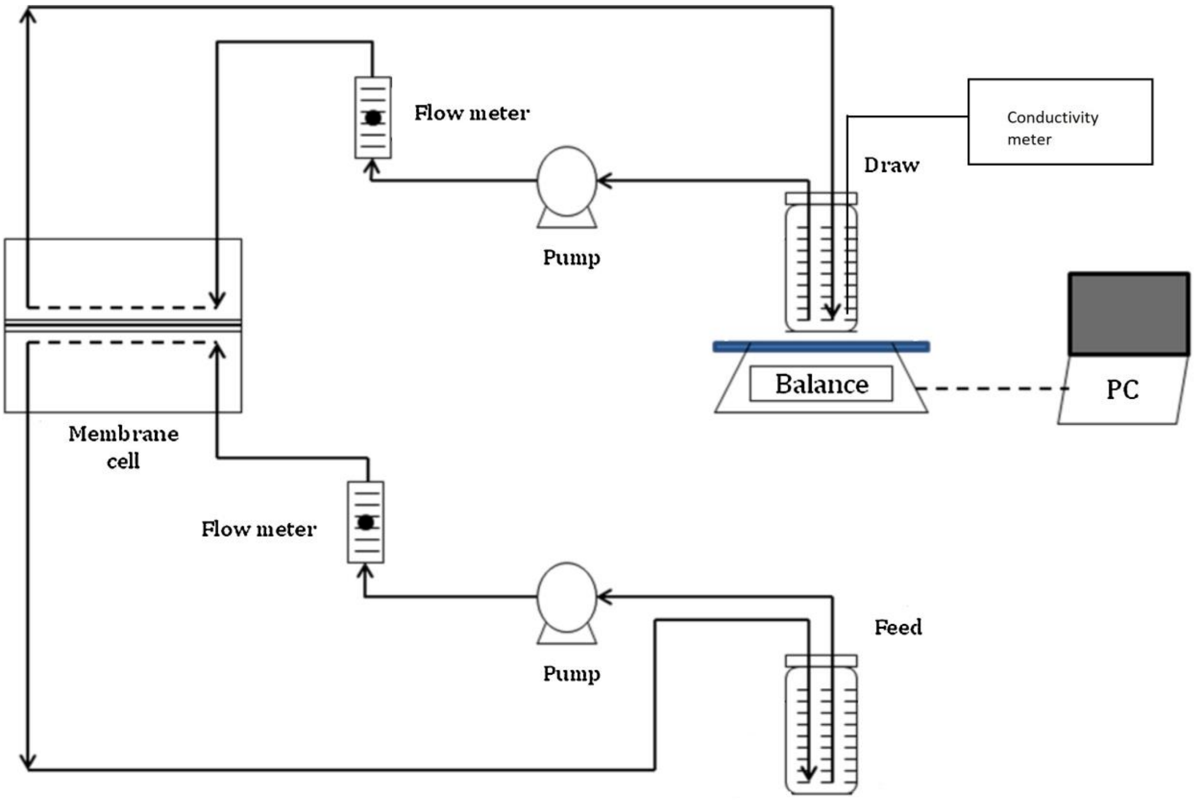

Figure 3.2: Schematic diagram of forward osmosis unit

3.8 Membrane Performance Study

In this experiment, the perimeter chosen to be studied was the concentration of oil in feed solution. Three different concentrations of oil were tested. For oil-in-water emulsion, the runs for each concentration were repeated twice using feed solutions of pure water and oil-in-water solution. Meanwhile, in the case of produced water, the experiments were repeated thrice for each concentration using pure water, sea salt solution and synthetic produced water solution. The water flux for each run was calculated. The fouling behavior of the membrane was also studied after the runs using scanning electron microscope (SEM).

3.8.1 Water Flux Calculation

The mass of water permeated to the draw solution was measured using the electronic mass balance every 5 seconds throughout the 1.5 hour run. The data obtained were directly transferred to the computer. Water flux is then calculated using the formula ;

$$J = \frac{V}{At} \tag{3.1}$$

where J = water flux ($L/m^2.hr$)

V = volume of permeate (L)

A = effective membrane area (m^2)

t = time taken for permeation (hr)

A graph of water flux against time was then plotted to evaluate the efficiency of the water separation.

3.8.2 Scanning Electron Microscope

Scanning electron microscope (SEM) was used to observe the surface of the membrane sheet before and after its use to detect the presence of oil molecules and cake layer formation on the membrane. Before SEM observation, the membrane was dried overnight. Then, it was cut into several small square pieces with an area of $1cm^2$. The square piece of membrane was then placed on the sample holders to have its surface analysed under the SEM. The same steps were repeated on membrane after the FO process. The surface before and after use were then compared in terms of their morphology to verify the possible deposition of oil droplets and formation of cake layer.

3.9 Membrane Rejection

Oil rejection of membrane can be determined with the help of the calibration curve. In the FO experiments involving oil-in-water emulsion and synthetic produced water as feed solutions, a 3mL sample of the draw solution before and after the runs were collected and had their absorptivity measured using the UV-Visible spectrophotometer. The difference of absorptivity between these two samples indicates the amount of oil that managed to pass through the membrane. By referring to the calibration curve prepared, the absorptivity value can then be converted into the form of oil concentration to determine the amount of oil present in the solution.

The oil rejection of the membrane can be computed using the formula below ;

$$R = \left(1 - \frac{c_p}{c_f}\right) x\ 100\% \tag{3.2}$$

where R = oil rejection percentage

c_p = average initial and final concentration of oil in draw solution

c_f = average initial and final concentration of oil in feed solution